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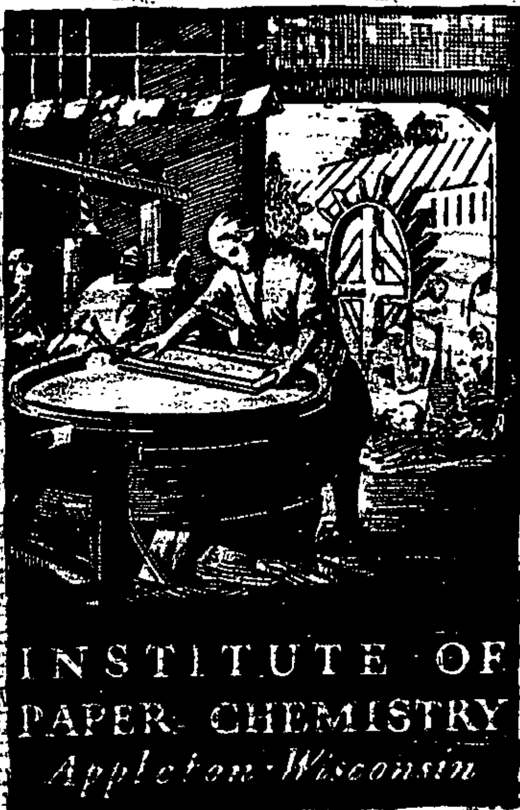

Meeting of

FOURDRINIER KRAFT BOARD INSTITUTE

AT

INSTITUTE OF PAPER CHEMISTRY

October 22-3, 1953

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Thursday Session

The use of chemicals in the packaging and food industries with particular emphasis on biphenyl in the citrus fruit field.

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Regulatory bodies and their procedures . . . . Hazleton

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THE USE OF CHEMICALS IN INDUSTRY

John G. Strange,
Vice President, The Institute of Paper Chemistry

Over the centuries mankind has been concerned, among other things, with the three basic requirements for survival--food, clothing and shelter. A great deal of our history is written around the various quests and systems which have been set up for the purpose of satisfying these requirements and it is interesting to observe that in our sociological studies today we continue to list these needs in their traditional order of importance, with food coming first. Certainly it seems logical to say that food always has been and will continue to be one of man's primary concerns.

In prehistoric days the procurement of food was reduced to its most primitive aspects. Early man killed his food and consumed it at once. His nomad society presented no problem of storing the food. He, of course, did not recognize the problem of preserving its flavor. He could not conceive of the possibility of improving its original nutritive value, and certainly there was no problem of transportation. His job at that time was simply to find his food and to eat it on the spot.

We have, of course, come a long way in the intervening centuries and especially have we made progress with respect to our food requirements in the last several decades. Our present organization of society is substantially based on the mechanisms which we have devised for storing and enhancing and preserving our various foodstuffs. This achievement is the result, of course, of many different areas of science and technology and the close teamwork of many different modern industries. Extremely important are the parts which the chemical industry and the packaging industry have played.

Everyone is familiar with the contributions which chemistry has made to the increase of food production and transmission. Certainly we would not be where we are today if it were not for the many insecticides, fungicides, and bactericides--to name only one area where chemistry plays a part. We have also the conservation of the natural color of canned fruits and vegetables by the addition of such chemicals as calcium hydrosulfide, sodium carbonate, calcium chloride and monocalcium phosphate. We have added, for example, small amounts of mono and diglycerides to hydrogenated vegetable oils, thereby improving the cake-making properties; antioxidants are used to improve the shelf-life and performance of fats and oils. One could go on for quite some time listing other ways in which chemicals are used by the food growing and food processing industries.

It is almost impertinent to suggest to this audience the part that the pulp and paper industry has played in providing mankind with his food requirements. Anyone who visits a modern supermarket can see very quickly the part that packaging has played in the protection and preservation of food. Someone has observed that the modern housewife practically lives out of the refrigerator and certainly this existence is possible only because of the part which your industry has played.

Our attainment of modern practices has not been simple and straightforward. Neither has it been entirely painless. It is quite obvious that the materials which are used for the preservation and storage of food must be screened very carefully to make sure that they do not jeopardize either the health or the bodily functions of the consuming public. Consequently, there has been a long research for chemicals which will either kill or retard the growth of fungi, bacteria, or insects, and at the same time present no hazard to the human species. In certain instances we have succeeded in finding chemicals with this selective toxicity. In other instances we continue to use chemicals which are highly toxic to humans, but various regulations have been established to insure their elimination before food reaches the consumer.

From a packaging standpoint, we have been concerned with this subject in several different ways. In the first place, packaging materials have presented an opportunity for incorporation of the various preserving chemicals. In other instances our industry has had to use various chemicals for the purpose of sterilizing the packaging material, thereby eliminating it as a factor of contamination, while protecting the packaged materials against further infestation. Finally, we have found that our industry frequently needs protection against fungal and insect life, and we have consequently used various slimicides and other materials in order to promote the more efficient manufacture of paper and in order to preserve machine clothing. The possible presence of these chemicals in our finished paper and their influence on foodstuffs may be a matter of concern.

As one views the future unfolding of the paper industry, he becomes impressed with the many opportunities which still exist in the packaging and transmission of foodstuffs. Certainly there are many additional commodities which might be wrapped or packaged in paper, especially as we find the ways and means of imparting new and improved properties to our packaging structures. It seems highly pertinent, therefore, that we concern ourselves with this general subject of chemicals in food packaging and that we utilize the experience which we are gaining in the citrus fruit field as a basis for understanding and implementing our growth in these other fields.

Although there probably would be some divergence of opinion among us as to the part that government should play in the regulation and administration of our society, I am sure that most, if not all, of us would concede that certain rules ans regulations are highly necessary in the field of public health and consequently in the use of chemicals for the preservation and packaging of food materials. Certainly there has to be some authority which is concerned with the various hazards which may be presented to the consuming public through the improper use of chemicals or through the improper distribution of foodstuffs in this highly specialized society. At any rate, whatever our philosophical opinions may be, it has long since been decided that this authority should rest with our various governmental structures and from the federal standpoint we have several important agencies.

In order that we may have some background on the position which these agencies have taken with respect to the use of chemicals in general, and their current position with respect to the use of biphenyl in particular, it seems appropriate that we spend a few minutes on the general subject of regulatory bodies and their procedures.

Dr. Hazelton will discuss this subject . . . .

REGULATORY BODIES AND THEIR PROCEDURES

Lloyd W. Hazleton, Ph.D.
Pharmacologist, Hazleton Laboratories,
Falls Church, Virginia

Since the time is limited, I have prepared an outline of some of the more important agencies because to read all, or even the pertinent parts, of the Acts which they administer would take more than our alloted ten minutes. If you can follow the white mimeographed sheets they will give you the different agencies with which we are primarily interested. It would be very interesting if we could go into the history and background of some of these agencies and their development of thinking but we will just not have the time. This is not a legal discussion of these laws; I am not a lawyer, just a pharmacologist. It is an attempt to try to interpret to you what these regulations mean in the way of collecting scientific data which are directed at two principal objectives: first, protection of public health; second, the allowance of progress in research and technology without undue retardation. So, going from the agencies and their authorizations which are on the paper, I would like to take up the scope of these agencies as a third topic.

Perhaps I should say, before I start talking about this, that under Secretary Benson's new reorganization plan some of these agencies are going to change as of November 1, but since this is still October this outline is, I think, intact and correct; therefore, what is presently called the Livestock Branch may change after October but the organization will be about the same. The Livestock Branch of the Department of Agriculture derives its scope primarily from the registration and labeling provisions of the 1947 Federal Insecticide, Fungicide, and Rodenticide Act. In Section III there is a provision which prohibits the sale of any fungicide, insecticide, or rodenticide, or what is generally termed an "economic poison" unless the label on that material is specifically approved by the Insecticide Division of the Livestock Branch. That Division now has some 50,000 labels approved and is setting up card indexes, using IBM machines, in order to keep track of them. The law further provides that those registrations must be renewed every five years. They are sending out postcards now. This Act was passed in 1947, became effective in 1948; 1953 completes the first five years. The process will be repeated each five years. The registration requirements are based, first, on specific proof of utility of the material; second, on the evaluation of safety of the material; and third, approval of a specific label. Note here that in the Act there is specific authority given to the Branch; that is, it says that the Director of this Livestock Branch administers that portion of the Act.

The Meat Inspection Division operates under a much older law, although it has been continuously revised. The Meat Inspection Act goes back to 1907. This Division operates primarily by a process which is known as a Standard of Identity procedure. Sausage, bologna, liverwurse, and bacon, for example, must meet certain standards of identity. These standards prescribe the amount of oatmeal or anything else that can be used as filler. Unless a meat or meat product meets its standard of identity it is in violation of the law. Then there are a group of materials which are allowed and are called "optional" ingredients. These ingredients do not have to be used in the meat product but may be included in certain quantities, and those quantities are very specifically outlined. These standards of identity apply also to additives which may be in meat products by reason of being present in containers which may permit the transfer of any chemical additive from the container to the meat. They apply to meat wrappers--anything which comes in contact with meat is susceptible or eligible for consideration under the Meat Inspection Act. Here again the approval by this agency is based on utility; that is, the additive must perform a function, it must be safe for the consuming public, and there must be suitable means for detecting each additive so that any regulation can be policed. Evidence of stability, of analytical methods, etc., must be provided by the manufacturer. This Act is again largely administered at the level of the Meat Inspection Division of the Department of Agriculture.

When we go to the Food and Drug Administration, which we are perhaps more interested in, under the Department of Health, Education, and Welfare, the procedures are multiple. They have one group of procedures which are based on adulteration; they have another group of procedures which are based on the establishment of tolerances, and then quite separate and apart--you see, this Act covers foods, drugs, and cosmetics--quite apart from that they have procedures based on certification comparable to those of the Livestock Branch and the Meat Inspection Division. Under the Food and Drug Act certification is specifically limited to certain classes of materials, such as certified dyes for foods, and insulin and antibiotics in the drug field. The Food and Drug Administration also operates under a fourth classification--a Standards of Identity. That finally got straightened out after years of hearings. The ice cream hearings are now, I think, being concluded. They set up a standard much like the Meat Inspection Division and there you have specification authorization.

Under the adulteration procedures there is no slip of paper which says this is or is not an adulterant. It comes under the very elusive term of "poisonous or deleterious." In essence, and if you will pardon my almost telegraphic style--I'm still watching the clock--Section 402 of the Food, Drug, and Cosmetic Act says that there shall not be present in a food any harmful or deleterious material, except that if it is not added it may be present provided it does not render the end food product deleterious. That is not very hard to interpret. It means that folks can sell mushrooms, or they can sell spinach which contains oxalic acid. By any toxicological test, oxalic acid would be declared strictly a poisonous or deleterious material. But no one adds oxalic acid to spinach and it does not render spinach harmful; therefore spinach can be sold. Mushrooms may also be sold, but not poison mushrooms because although the poison is not added it renders the final food poisonous. That is Section 402, and there usage is taken into consideration.

Section 406 has essentially the same wording except that it says that any poisonous or deleterious material added to any food shall be deemed to be unsafe, except where such substance is required in the production thereof or cannot be avoided in good manufacturing practice. Then it says "but when such substances are so required or cannot be so avoided, the Secretary shall promulgate regulations limiting the quantity therein or thereon to such an extent as he finds necessary for the protection of public health." Here the question that is basic, therefore, is not whether the additive renders the end food product deleterious but whether the material going into the end product is poisonous or deleterious. In that particular instance usage cannot be considered, and that is where so many people err in considering these problems; they say, "Why, to be specific, with biphenyl, you would have to eat 40 pounds of oranges," or something to that effect. That is not the question. Under this Act, if the material added is poisonous, it doesn't make any difference how many oranges you would have to eat. I think that this is basic in the problems that we who are faced with the study of this question all the time some day hope to see clarified. Unfortunately, the proposed new amendment is not, I think, going to clarify it one little bit.

Another interesting point about this Food and Drug Law is that it does not mention anything lower than a Secretary (Mrs. Hobby right at present). It says you shall submit to the Secretary various evidence as required. I think a lot of people have been led astray in trying to get their answers by going too high in the Federal Security Agency, or the new Department of Health, Education, and Welfare. For all practical purposes this law is administered exactly like the others in that in the Food and Drug Administration the group responsible for determining safety or hazard is the Division of Pharmacology, just like we have the Livestock Branch or the Meat Inspection Division in the Department of Agriculture. Most of our problems involved in these questions are answered at the Division level, but people take them anywhere from the Commissioner on up to the Secretary of the Department and are quite confused by not going back down to the people who actually execute the law, as they would if this law were written a little bit differently. It has led to untold confusion because they don't realize that the Secretary or even the Commissioner of Foods and Drugs is not the person who really is guiding the policy and determining the adequacy of tests; it is the Pharmacology Division. This Division passes on Section 402 and Section 406, whether it is foods, or drugs, or cosmetics. Without an understanding of the operations of the Pharmacology Division, no one is going to get far in talking with Madam Secretary because she is not spending her time trying to interpret Sections 402 and 406.

I believe that uses up my time. I would like to point out, however, that in all of these three laws that I have mentioned, the ultimate interpretation depends and is based on the evaluation of experimental, biological data. We cannot use, in this country, humans for all of these tests. Animal experimentation is the primary basis for determining whether these products and additives are harmful or deleterious or whether they are safe. And that aspect of research work is to be discussed by Dr. Willis Van Horn, who is a group leader in biology here at The Institute of Paper Chemistry.

THE NATURE OF BIOLOGICAL RESEARCH

Dr. Willis Van Horn
Research Associate, The Institute of Paper Chemistry

This discussion is concerned with first the character and nature of biological investigations, and second, the character and nature of one of the branches of biology—toxicology.

Biology is the science of life, it is the study of living things. One of the fundamental characteristics of living matter is its variability. There is variation between individuals of a single species, and there is variation between species. This variation is manifest in a multitude of ways. It may occur in the normal metabolic reactions within the body, it may occur within an organ system, or it may occur within individual cells. Furthermore, variation may occur in the reaction of different individuals to the same external stimulus.

This variation has a great influence on the nature and technique of biological research. It is not possible for the biologist to work with the frame of mind, attitude and precision that characterizes most of the physical sciences. Adaptations in technique must be made for changing conditions, and compensation must be made for the high degree of variation. This compensation is obtained by using a large number of individual tests and from them establish a trend. In this area the statistical approach is useful.

It is in the reading of results that the highest degree of skill, insight, experience and understanding is required. To the uninitiated it is very easy to go astray and arrive at a conclusion which is unwarranted by closer examination of the data.

In the light of these facts about biology in general let us examine the science of toxicology, at least that part of it that concerns us here.

As we all know, before any new medicine, drug or the like can be used on human beings it must be first tested to see if it will cause any effects, ill or otherwise, other than those for which it was intended. Similarly when a chemical is incorporated into food designed for human consumption, its manufacturer, or the food processer, would as a matter of course, determine if that chemical, in the amounts taken by people under the circumstances of processing, would be deleterious to health.

From the standpoint of pure science the best way to determine this would be to get a bunch of people together and feed them varying amounts of the chemical—amounts determined by the use level--and see what happens to them. This is obviously impossible so the toxicologist must use laboratory animals, the most common of which are white rats, dogs, monkeys, and sometimes guinea pigs and rabbits.

A number of healthy animals are chosen and divided into two groups. The first group--the experimental animals--are fed a balanced diet plus the chemical in question in varying dosages. The other group--the controls--are simply fed the balanced diet. As the experiment progresses observations are made for gross external effects such as weight, condition of coat, etc. After a suitable period of feeding both groups are sacrificed, gross examination of internal organs made, and then microscopic sections of such organs as the kidney, liver, spleen, etc. made. Comparison of the preparations from experimental animals with those from the controls will, in theory, at least, reveal evidence of toxicological or pathological conditions.

In addition to the animals killed for microscopic studies another group is used to determine the effect of the chemical or reproduction. Thus the experimental and control animals are brought into appropriate procreational proximity within their respective groups, and the number and character of offspring observed.

By a series of such experiments it is possible to determine the lowest dosage rate of the chemical from which there is no evidence of toxicological or pathological change in the experimental animals. This level is then translated back to the use level of the chemical to be used in food and to the anticipated intake of the chemical by human beings in using the food. Here is one of the most critical areas of all types of pharmacological and toxicological research, namely, the interpolation from laboratory animals to human beings. The assumption must be made that humans will react to a given chemical in the same manner that rats, dogs, or other animals will react. This assumption may not always be justified. If the human intake is below that which was established on the animals then it may be concluded that the use of the chemical on foods is without hazard to public health. If, on the other hand, the human intake is above the safe level established on animals, the general conclusion is that the chemical should not be used.

As was pointed out earlier the very character of biological research imposes a number of requirements on toxicological investigations. In the first place this type of investigation requires the services of a competent toxicologist. He will be able, first of all, to distinguish between changes which come about through natural causes and those which are a result of the chemical. Variation being what it is, this requires long experience, insight and good judgment.

Another requirement is proper design of experiment. As has been said these experiments may take as long as two years. A badly designed program therefore could easily result in loss of time and money.

One of the more perplexing aspects of this type of investigation is the problem of how it can be controlled. The controls, it will be remembered, are the ones which received everything but the chemical. Cases are on record where most of the control animals died but the experimental animals survived. Theoretically when the control animals die the experiment is ended. Actually, however, in view of the high cost in terms of time of work of this type, dead control animals are replaced by live healthy ones. On the other hand if an experimental animal dies it is concluded, usually, that the cause of death was the chemical and the death so recorded. One point that seems to be missed is that when control animals die, say from some respiratory infection or the like, and the experimental animals do not, it is altogether possible that the chemical in the cells of the experimental animals is acting as a medicine to protect them and thus as a protective rather than a harmful agent.

Notwithstanding vagaries of biological investigation, there is no other way in which a problem in toxicology can be solved. And I don't mean to give you the impression that biological investigation is bad or uncertain; what I do mean is that, to be effective and significant it must be done with good judgment, common sense and understanding.

THE USE OF BIPHENYL IN CONNECTION WITH CITRUS FRUITS

Elmer P. Wheeler,
Industrial Hygienist, Monsanto Chemical Company

My remarks concern two aspects of the use of biphenyl—first the reduction of spoilage in citrus fruits; second the uses of biphenyl in some of the other better known applications of the chemical industry. I would like also to present a brief summary of the events leading to recent government concern over the specific application of biphenyl in the citrus industry.

In relation to biphenyl itself, it is of interest to point out that this material is an organic compound made up of two benzene rings. Its production is a matter of straightforward chemical reaction, the raw material being benzene which is heated over a molten metal catalyst at which time the two rings join with the liberation of a molecule of hydrogen. The biphenyl itself is a colorless crystalline material at normal temperatures with a melting point of 69-70°C. or approximately 158°F. It has an aromatic odor which is quite characteristic, similar to some phenolic compounds and to the citrus consuming public it might best be described as medicinal. This product is insoluble in water but is readily soluble in alcohol, ether, benzene, and most common organic solvents.

As an industrial chemical, biphenyl is a raw material or intermediate in the production of chlorinated biphenyls, hydroxybiphenyls, nitro- and aminobiphenyls, and biphenyl oxide. In these end products it may be used as a hydraulic fluid, a heat transfer agent, in the covering of electrical cables, as a plasticizer in the plastic goods industry, in synthetic paints, and as a dye intermediate. The production and handling of biphenyl in these fields has been singularly free of any incidence of toxic effects on the workmen in chemical plants. This is probably true for several reasons. In relation to many other commonly handled chemicals, biphenyl is not particularly toxic; secondly, its vapor pressure at room temperature is very low and normally, only small amounts of the material will be present in the atmosphere. In addition, when the material is heated to a temperature where it does volatilize, inhalation of vapors results in irritation to the respiratory tract and eyes and normal industrial practice is to eliminate or control the vapors, either by using a closed process, by mechanical exhaust ventilation or by providing respiratory protection for the workers.

You may comment justifiably that this experience in the chemical and other fields is not particularly pertinent to the use of biphenyl in connection with citrus fruit. It is true that there can be little correlation between the exposure in our plant or in the plants of our other industrial chemical customers—where ingestion or oral intake of the product is certainly a rare exception—there can be little correlation between this type of potential exposure and the type of exposure where biphenyl becomes incorporated in food products. On the other hand, there have been many cases in the past where just this type of satisfactory experience in industry plus a minimum amount of animal experimentation which Dr. Van Horn mentioned previously, has served for a basis for approval of materials in food applications. I would like to mention that one other point in talking to you about these other uses is the fact that in these uses the production and consumption of biphenyl is in the thousands of tons annually. To the best of our knowledge, the current úsage in the citrus industry represents something under one or two hundred tons per year. I will not predict what the potential is for this market but at the present time the use of biphenyl in citrus applications is a very small proportion of the total biphenyl use.

Biphenyl to reduce decay of citrus fruits during shipping is a relatively recent development and without going into detail as to the names of the scientists involved, the places where research was done, or the patent situation, I would like to mention some of the pertinent facts. The discovery that biphenyl aided in the preservation of citrus fruits was made in England in 1935 and shipments of oranges in biphenyl-treated wraps began arriving in England in 1938 from Palestine. We've been told that shortly thereafter the odor of biphenyl emitted in these shipments was brought to the attention of public health officials in Great Britain, who promptly forebade further entry of such shipments. This action was taken without any investigation as to whether or not the use of biphenyl introduced any hazard to the consuming public. Despite this prohibition, which has never been rescinded, citrus in biphenyl-treated wraps is still being admitted to Great Britain. We don't know of any explanation for the apparent lack of enforcement on the part of the authorities other than perhaps the fact that shortly after this banning of shipments, England got involved, very early, in World War II and there was almost complete cessation of shipments of citrus fruits to the British Isles for the duration of the War. Then about the end of the War there were publications in England which I think have some bearing on why authorities close their eyes to the entry of citrus in biphenyl-treated wraps. I don't think that Dr. Hazleton would particularly agree with me on this, but I do think it's an example of the thinking at that time. It isn't the thinking of this country in 1953.

The animal toxicity data that was published in 1945 covered four weeks of feeding to laboratory rats. The pathology on these animals showed no changes in the animals on the basis of that amount of feeding; and correlating the results of the feeding tests with the maximum amount of biphenyl which was found in a few oranges at that time, it was stated and I quote "A man eating about 400 oranges a day would still not be getting enough preservative to do him any harm." I repeat that I still believe that this type of data carried a lot of weight in Great Britain and I am also convinced that it meant a great deal to the people in this country during the early developmental stages of the use of biphenyl. There were confirmatory data published in 1947 as a result of some work sponsored at Kettering Laboratories by my company, and I know that the results of these experiments served to allay any fears that the application might be unsafe. Unfortunately, it's just a question of the animals not having received a dosage over a long enough period of time to develop the pathology that has appeared in more recent tests.

The events which lead us to the present concern over biphenyl indicate that the origin might be traced to about the middle of 1950 when my company received a request for information from Israel, the request asking if we knew of any basis for a rumor that biphenyl could cause cancer. I shudder to mention this because I don't want anyone to carry away from this meeting the fact that I or anybody else suspects with any justification that biphenyl causes cancer. As a matter of fact, in our efforts to track down this rumor we have been unsuccessful in finding any basis for this statement either in this country or anywhere else in the world. While we were trying to learn why such a rumor could or should have arisen, we began to get inquiries from people in this country asking for the basis of this rumor. And it was shortly after that when we learned that two-year feeding tests were to be undertaken at Stanford Research Institute. I don't know which came first--whether a skin painting test which is a test for determining carcinogenic potential was started before the two-year feeding tests were started or not, but the two investigations are related, I believe.

I think many of you are familiar with the present predicament over the use of biphenyl for citrus fruit shipping containers, but I'll take time to review it. As the result of the two-year animal feeding tests which were conducted by Stanford Research Institute, the Food and Drug Administration has indicated that they believe biphenyl is a poisonous and deleterious substance. The action which they would normally consider next, that is, action to indicate whether or not biphenyl can be continued in use, has I believe been delayed much longer than Dr. Hazleton and some of those who have been more closely associated with toxicological affairs would have believed possible. The data that came out of the Stanford Research Institute report indicated that at certain levels there was pathology in experimental animals. Whether or not this pathology is significant when one compares the levels to which the animals were exposed with the level or dosage a human being might be exposed to is still open to question. With many questions raised by the S.R.I. report still unanswered, I don't think any of us would quarrel with the action of the Food and Drug Administration--particularly if one imagines himself in the position of the Administration who is charged with safeguarding the health and welfare of 160 million people.

One of the other points raised by the S.R.I. report and by the F.D.A. pertains to the residue of biphenyl on or in fruit shipped in biphenyl-treated containers or wraps. If biphenyl is to be used, then there has to be available a reliable method or methods for the detection of residues in the food product. Monsanto Chemical Company has been co-operating in research on such methods and the results of this research are to be discussed by our next speaker, Mr. Edward Dickey of The Institute of Paper Chemistry staff.

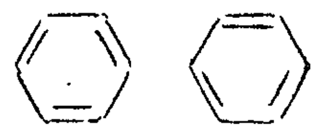
PROBLEMS OF DETECTION AND MEASUREMENT OF BIPHENYL

Edgar E. Dickey,
Research Assistant, The Institute of Paper Chemistry

BIPHENYL

Colorless crystals
m.p. 70°C. (158°F.)

$C_{12}H_{10}$



b.p. 256°C. (493°F.)

Odor: characteristic

Vapor pressure: 4 mm. at 100°C. (212°F.)

0.04 mm. at 25°C. (77°F.)

At the outset two facts seemed to sharpen the problem of accurately measuring the amount of biphenyl in citrus fruit: (1) The amounts of the agent, based on existing knowledge, would lie in the range of 0 to 100 p.p.m. and (2) none of the methods was absolute (required no blanks).

Because most of us are conversant with the expression parts per million (p.p.m.) for insecticides, fungicides and the like, this mode of expression has been adopted for the biphenyl work. The problem required that we devise a method which would be practical in terms of time, manpower, and equipment, and which would be—if possible—an absolute method. If <u>one part</u> is <u>one orange</u>, a million parts would occupy ten refrigerator cars of 1000 boxes each. Stated in another way, if <u>one part</u> is <u>one milligram</u>, then beaker No. 1 holds one million parts of oranges, and beaker No. 2 holds the pulp and No. 3 holds the peel from one million parts of this type of orange. In the next exhibit, the smaller ampule contains 2 p.p.m. and the larger one contains 50 p.p.m. based on the whole fruit (beakers 1, 2, and 3).

POSSIBLE METHODS OF ANALYSIS

A. Chemical

B. Biochemical

C. Physical

1. Weight
2. Odor
3. Color
   a. Infrared
   b. Visible
   c. Ultraviolet

A. Biphenyl contains (1) no "odd" element (only carbon and hydrogen), and (2) is chemically less reactive than nearly all other organic components of citrus fruit. These facts eliminate the possibility of devising a practical chemical method of analysis. However, an older method, based on a chemical reaction, is reported with an accuracy of $\pm$ 20%; it has no usefulness in the present problem and little hope for improvement.

B. Biochemical analyses are rarely rapid enough for routine work. Besides, the general lack of knowledge of the biochemistry of biphenyl precluded the development of such methods of analysis.

C. 1. Amounts are too small for practical separation and weighing.

2. Biphenyl has a characteristic odor to which the nose is very sensitive. The problem is one of removing masking and interfering substances and of calibration.

3. a. Because biphenyl has a characteristic absorption band at 14.34μ one method is based on this property. To the best of our knowledge the method is not absolute since a background or blank value contributed by the orange oil must be subtracted. Also, infrared instruments are more costly and not commonly available.

b. Biphenyl is colorless. A chemical reaction to form a colored substance would be required and this possibility was eliminated under point A above.

c. Biphenyl has a very strong absorption of ultraviolet light at 248 mmu. The intensity of absorption at this wave-length and the general availability of ultraviolet instruments brought the research to a focus at this point.

It had been recognized by other workers that measurements of biphenyl with a ultraviolet spectrophotometer were feasible. But we found it necessary to improve the existing procedures of sample preparation so that all interfering substances were eliminated.

The method as now employed was suggested by Monsanto and developed by Monsanto and the Institute. It takes advantage of two characteristics of biphenyl: (1) its considerable volatility with steam and (2) great chemical stability of biphenyl compared with the orange components. Point (1) separates biphenyl from the bulk of the orange which is not volatile, and point (2) chemically removes the interfering components which are volatile. A novel adaptation which utilizes point (1) but substitutes a "chromatostrip" for the chemical operation is in use in California. It is a relative method since the human eye must be calibrated for quantitative measurements.

HOLDING AND SHIPPING EXPERIMENTS

R. C. McKee, The Institute of Paper Chemistry

As was brought out earlier in the panel discussion, when the Food and Drug Administration rendered its decision regarding the classification of biphenyl there was no adequate method available for accurately determining small quantities of biphenyl in oranges. Likewise there was available no reliable data regarding the amount of biphenyl which an orange would pick up during shipment or storage in fibreboard boxes. Mr. Dickey has described the problems involved in developing a method. I would like to describe the holding tests which were designed to put these methods to work in the gathering of data regarding the biphenyl content of citrus fruit because it is important that we know the biphenyl residue level of fruit packaged in treated fiberboard boxes in order that we may determine whether or not public health is being endangered.

At the meeting held at the Institute on April 6, 1953, two sets of holding experiments were set up which we will refer to as controlled holding tests and consumer tests. The controlled tests were set up for the purpose of determining the biphenyl content of citrus fruit packaged in treated paperboard boxes as a function of time and temperature of storage under controlled conditions of relative humidity. The holding tests as designed at this time are illustrated in Figure 1.

The consumer tests were set up to determine the biphenyl content of commercial packs of citrus fruit, transported by normal carriers, both upon arrival at destination and after normal shelf exposure. The general procedure being followed is illustrated in Figure 2.

When the holding tests just described were initiated they were undertaken with the expectation that the Stanford report would specify the maximum level of biphenyl intake that the experimental animals used could safely tolerate. This level could then be weighed against the residue levels found in the holding tests. However, the Stanford report in its final form did not specify the maximum level which could be tolerated. However, sooner or later such data will be available to the F. D. A. which will permit them to establish tolerance limits, then the data from these holding experiments will be essential.

Figure I.

CONDITIONS OF CONTROLLED HOLDING TESTS

1— Participating Laboratories

- a — Citrus Experimental Station, Lake Alfred, Florida.
- b — U.S. Dept. of Agriculture Pasadena, California.

2— Fruit

- a — Oranges (Florida and California)
- b — Lemons (California)

3— Storage Condition

- a — Temperature
  - (1) 35—40° F.
  - (2) 70—75° F.
  - (3) 95—100° F.
- b — Humidity — $80 \pm 2\%$ RH
- c — Storage Time
  - (1) 1 wk
  - (2) 2 wk
  - (3) 4 wk
  - (4) 8 wk
  - (5) 16 wk

4 — Testing

- a — 3 boxes per condition (see 3—c above)
- b — 2 oranges per box adjacent to Treated panel.
- c — Pulp and peel to be analyzed separately.

Figure 2.

CONDITIONS OF COMMERCIAL PACK TESTS.

1— Participating Laboratories

a — Monsanto Chemical Co.

b — Institute of Paper Chemistry

2— Fruit — Oranges

a— Florida

b— California

3— Method of Transportation

a— Carload (RR)

4— Testing

a— 11 boxes per car (Taken from selected locations in car)

b— Evaluated for biphenyl content of peel and pulp

(1) On arrival — 2 oranges from each box, one orange adjacent to treated panel and one adjacent to untreated.

(2) After 9 days of exposed storage at 60—70°F.

SUMMARY OF DATA COLLECTED BY MONSANTO AND THE INSTITUTE OF PAPER CHEMISTRY

Dr. John Green,
Research Associate, The Institute of Paper Chemistry

I would like to discuss some consumer experiments made by Monsanto Chemical Company and The Institute of Paper Chemistry. We have been interested in the amount of biphenyl in oranges at the consumer end, when the housewife buys at the corner grocery store. Therefore a series of 18 shipments of biphenyl-treated oranges were sent from California during the summer to both Dayton and Appleton at regular intervals. Each shipment consisted of 12 cartons, containing 42 pounds oranges each, were spotted in freight cars and forwarded to us by express after the cars were opened, generally in the vicinity of Dayton or Chicago. Approximately two weeks elapsed from time of shipment until the cartons were opened for sampling.

The liners in each carton were analyzed for residual biphenyl content. Samples of 2 to 16 oranges were taken from each carton and either the whole orange analyzed, or peeled carefully and the pulp and peel analyzed separately.

The first chart shows the data obtained by Monsanto Chemical Company, this work being done by Dr. Johns and Dr. Beasecker in Dayton. The ordinates are (a) number of shipment, given chronologically, and (b) the biphenyl content of peel and pulp, in parts per million based on the fruit weight, and the biphenyl content of the liner given in milligrams per square inch. All values for each shipment are averages for the 12 cartons. The several graphs are displaced to show similarities in profile.

The average values for all 12 shipments are 24 p.p.m. for the peel, 0.8 for the pulp, and 0.5 mg./sq. in. for the liner. (3 mg./sq. in. is equivalent to 1 lb./1000 sq. ft.).

It is interesting to note the close relationship of the three sets of data, especially the amount of biphenyl left in the cartons at time of sampling. We have no data for the biphenyl content of the liners at time of shipping.

The second chart shows similar data obtained here at the Institute, the work being done by Messrs. Dickey, Pearl and myself. Here the data are a bit higher than in the first chart, the average for all shipments being 37 p.p.m. for peel, 0.60 for pulp and 1.77 mg./sq. in. for the liner. So these values are of the same general order as those of Monsanto. Dr. Newhall of the Florida Experimental Station and Dr. Beavens of the U. S. Agricultural Station at Pasadena report similar data.

Tho data presented so far represent 2 to 16 oranges per carton or 24 to 192 fruit per shipment. What is the variation between single oranges in a carton? Are we taking sufficiently large samples to get representative samples at a 4-fruit or a 8-fruit sample level? Well, in the third graph are shown the results of analyzing every 4th orange in a carton of 160 oranges. The bottom graph represents the biphenyl content of whole oranges selected from the center of the box, not touching the liner. The center graph represents the oranges in contact with the liner and the top graph gives the over-all picture. It is obvious that a sample of one or two oranges can give a value ranging from 6 to 20 p.p.m. In future work it has been decided to take a sample of 30 oranges from a box.

The lower biphenyl content of the center oranges in contrast to those in contact with the liner has been noted again and again in this work, both by Monsanto and by the Institute. This is for a 2-week storage period. In contrast, the people at Florida and Pasadena report, for storage periods of 4 weeks or more, no differences at all for these two positions within the carton. This may be explained in part by the static storage in the latter case, in contrast to the agitation of cartons during shipment.

A few shelf life experiments have been run, where the oranges were exposed to the open air for 7 to 14 days, as would occur in a retail store. In this time little or no dissipation of biphenyl has been noted, other than the loss of biphenyl odor from the surface of the fruit.

In conclusion I would like to point out that while I do not know whether they raise many alligators in California, they do raise a lot of oranges out there. We analyzed a few of them. We sorted 20,000 oranges in all, peeled 1500, and made 500 extractions and ultraviolet absorptions during this work. We did leave a few fruit out west however, and now Dr. Beavens, of the U. S. Agricultural Station at Pasadena, California, will tell you what he has been doing with them.

BIPHENYL RESEARCH UNDER WAY IN CALIFORNIA

E. A. Beavens
U. S. Department of Agriculture, Pasadena, California

Dr. Hazelton has given you an excellent discussion of the functions carried on by various regulatory agencies established by the Federal government to control the use of chemicals in food industries. Several of these are in the U. S. Department of Agriculture. I would like to point out, however, that the Department also has a number of research agencies, of which our Pasadena Laboratory is a part, whose job it is to carry on agricultural research work to produce new or improved food products and develop methods to deliver them to the nation's markets, either fresh or processed, with minimum loss of quality or nutritive value. These research agencies have no regulatory duties to perform; they are service groups set up to aid the farmer and allied workers in the food industries.

In recent months our laboratory has been co-operating with the Institute of Paper Chemistry, Monsanto Chemical Company, the Florida Citrus Experiment Station, and agencies in California, on the use of biphenyl for controlling mold decay in citrus fruits shipped in sealed fiberboard cartons. The use of this fungistatic agent is particularly important to citrus growers in California-Arizona since they ship approximately 79 percent of their Valencia and Navel oranges, lemons, and grapefruit as fresh fruits. This represents an average of approximately 390 million boxes (wood crates) shipped per year, valued at around 160 million dollars. In contrast, Florida processes approximately 65 percent of its citrus crops, largely in the form of frozen concentrates which are becoming increasingly popular each year. California's problem is to ship fresh citrus fruits distances up to 3,000 miles in this country and longer distances overseas, and biphenyl appears to aid in helping deliver this fruit to distant markets in the best possible condition.

The use of biphenyl for preventing molding of citrus fruits in transit has important economic significance. In past years California and Arizona have shipped citrus fruits in standard wood slat crates. Use of these containers involves expensive handwrapping and hand packing individual fruit, and with ever-increasing waves for this labor the costs of handling have been mounting steadily. Something had to be done to reduce excessive packing costs, and the fiberboard half-box appeared to offer possibilities. Unwrapped lemons, volume-filled into fiberboard containers were the first citrus fruits tested in California. It soon became evident that mold decay increased in fruits packed in sealed cartons, and the use of some fungistatic agent would be necessary to reduce losses. Biphenyl had been used successfully in other citrus-producing countries, so it was a natural choice for use in this country. In addition to shipping lemons in biphenyl-treated cartons, some California Velencia oranges were shipped in this manner during the past season, and the industry is planning to test biphenyl on Navel oranges and grapefruit during the coming season.

The citrus industry in California has shown an enthusiastic interest in the biphenyl problem. A "Citrus Industry Committee on Biphenyl" was organized last spring consisting of leaders in the fresh fruit marketing organizations. In turn this committee appointed a technical subcommittee to plan and conduct co-operative research on biphenyl and related problems. Both committees have been active in organizing research work in this problem area to accumulate technical data related to economic necessity, storage experiments, biphenyl absorption tests, toxicity tests, new masking agents, use of different cartons, development of biphenyl substitutes, etc. I would like to describe briefly how this co-operative work is being handled.

The following agencies in California are participating in these co-operative studies. Department of Agriculture agencies include the Division of Handling, Transportation and Storage Laboratory at Pomona and the Fruit and Vegetable Chemistry Laboratory at Pasadena; state agencies include the Department of Plant Pathology of the University of California Citrus Experiment Station at Riverside, Department of Agricultural Economics and the School of Medicine of the University of California at Los Angeles; industry groups include local fiberboard carton manufacturers and citrus fruit marketing organizations.

The USDA laboratory at Pomona has worked for many years on the handling, transportation and storage of citrus fruits, so their work has included packing fruits in biphenyl-treated cartons and storing under controlled conditions of time, temperature and humidity simulating commercial practices. At the end of each storage period, which usually lasts up to four weeks, the fruit is examined for mold decay and other defects, such as loss of stem-end buttons, which may adversely affect fruit quality. Our laboratory has been analyzing these test lots of fruit for their biphenyl content so the results may be correlated with those obtained by the Institute of Paper Chemistry and Monsanto Chemical Company on the biphenyl content of fruit shipped to these laboratories from California. In addition to this type of work, the Pomona Laboratory has been studying the precooling of citrus fruits in closed cartons. Fruit temperatures in sealed cartons were checked in several packing houses, and in some cases temperatures as high as 96°F. were recorded during the precooling process. The fiberboard cartons fitted with biphenyl-treated collars acted as good insulating material which prevented escape of the heat of respiration generated by the enclosed fruit. Work is now under way to study the optimum conditions for proper stacking of cartons in refrigerated cars to allow for maximum cool air circulation through the stacks.

The Pasadena Laboratory has been working on the development of an analytical method for the quantitative determination of biphenyl in citrus fruits and fruit products. A method has been perfected which can be used in the analysis of both fruit and fiberboard collars for their biphenyl content. Many lots of citrus fruits have been analyzed with this method to determine amounts of biphenyl absorbed by the whole fruit, peel, and juice under known storage conditions. In general, it was found that the higher the storage temperature the more biphenyl will be absorbed by the fruit. Biphenyl-treated fiberboard collars manufactured in the Los Angeles area have been analyzed, and they were found to vary from almost no biphenyl to the recommended 4 pounds per 1000 square feet of surface. The results indicated that better control methods are needed to produce collars of uniform biphenyl content, and considerable biphenyl is lost during the flat storage of treated collars.

Citrus fruits are a primary source of our daily requirement for vitamin C, so it was natural that the Food and Drug Administration would be concerned regarding any adverse effects of biphenyl on this important vitamin. We have tested the storing of citrus fruits in contact with biphenyl for long periods, and we are glad to report no adverse effects on the vitamin C content. We have conducted numerous taste tests on juices prepared from biphenyl-treated citrus fruits to determine what levels of biphenyl can be detected. Several members of our panel can detect as low as 0.5 p.p.m. biphenyl in lemonade, although a majority cannot detect it until the level is raised to 5 p.p.m. When pure biphenyl, without any masking agents, is added to orange juice its presence cannot be detected until the amount reaches around 10 p.p.m., so we suspect the masking agents are part of the off-flavor associated with biphenyl. Preliminary experiments have been conducted on the use of various types of household juice extractors on the biphenyl content of citrus juices. Power driven and hand reamers are about equal in the amounts of biphenyl they add to juices, while the press types add twice as much. Most of the biphenyl absorbed by citrus fruit is located in the outer oil-bearing layer (flavedo), and the more this layer is macerated the more oil, and consequently more biphenyl, is added to the juice. Other experiments have been included to determine effects of processing on the biphenyl content of products made from treated fruit. For example, how much biphenyl is lost when treated oranges are made into marmalade or frozen purees.

Dr. L. J. Klotz, Head of the Department of Plant Pathology of the University of California Experiment Station at Riverside is co-operating in this project to test other chemicals which can be used to prevent mold decay in citrus fruits. Dr. Klotz is a recognized authority in his field, and we fully expect he will come up with a good substitute for biphenyl. In fact, he already has several leads which look promising. He has two full-time scientists on this job, and we understand part of this work is financed by FKI.

The School of Medicine of the University of California at Los Angeles has been conducting tests on the application of biphenyl on the skin of mice and other test animals. Dr. W. A. Selle, who is in charge of this work, has even applied biphenyl to his own skin for prolonged periods without harmful effects. In addition to this technical work the Department of Agricultural Economics of the same institution is accumulating data and statistics for the preparation of a comprehensive report on the economic necessity of using biphenyl in the citrus industry. This report, plus a similar one being prepared in Florida, should help establish the necessity of biphenyl in helping preserve citrus fruits shipped in fiberboard cartons.

The Research Department of Sunkist Growers of California is carrying on some interesting taste tests on juices and food products made from biphenyl-treated citrus fruits, and I am sure everyone here would like to be a member of this taste panel. Our laboratory has co-operated in this work by storing citrus fruits in treated cartons to build up the biphenyl content to known amounts. The fruit is then turned over to the Sunkist Research Department where it is made into juices, ades, pies, cakes, and other products, including a twist of lemon peel in Gibson cocktails. These products are tasted, using several methods, to determine if biphenyl can be detected, and at what levels. Biphenyl-treated oranges are packed in lunch boxes with different sandwiches to see if they will pick up off-flavors, and biphenyl-treated citrus fruits are stored in household refrigerators to see if they impart off-flavors to butter and other products.

Local fiberboard converters have been co-operating in this work by supplying cartons and biphenyl-treated collars, pads, and liners. They have been particularly helpful in preparing collars containing different amounts of biphenyl from the recommended 4 lb./1000 sq. ft., to as low as 0.5 lb./1000 sq. ft.

SIGNIFICANCE OF AVAILABLE DATA

Dr. Willis M. Van Horn,
Research Associate, The Institute of Paper Chemistry

I think we can summarize our present position with respect to biphenyl residues of oranges shipped in biphenyl-treated fiberboard containers very briefly. But before that is done, I feel that some mention should be made of all of the people and organizations who have co-operated on this work. Among them are Monsanto Laboratory at Dayton with Dr. Johns and Dr. Baesecker, the California group with Dr. Beavens, and the Florida group with Dr. Newhall. All of us are especially indebted to Mr. A. F. Mooty of F.K.I. and to Messrs. W. E. Baier, D. M. Anderson, and J. MacRill of the Sunkist organization.

The first important contribution to this study has been the development of satisfactory methods for the accurate measurement of biphenyl in parts per million. Such methods are essential to us in our work, and they will be required by the Food and Drug Administration when and if tolerance limits are set.

We have completed our studies on biphenyl residues on oranges shipped from California, and will expect to complete similar studies on Florida oranges this fall. We will also want reference information on other citrus fruits.

These data are absolutely necessary for the final resolution of the biphenyl problem when the time comes to establish tolerance limits. When such limits are finally set, probably after additional toxicological studies have been made, they will be the reference point from which calculated human intake of biphenyl can be made. That, of course, will be of paramount importance to the regulatory agencies.

It is my pleasure to introduce the next speaker, Mr. G. J. Ticoulat, Vice President, Crown Zellerbach Corporation, San Francisco.

PRESENT POSITION OF PURE FOOD AND DRUG

G. J. Ticoulat,
Vice President, Crown Zellerbach Corporation

There has been a great deal of erroneous information passed along as to our reasons for deciding to sponsor the research program at Stanford to determine the toxicological properties of diphenyl. In 1950, at the time we decided to have this study made, the use of fibre boxes in the citrus industry had not been given serious consideration by the packers or growers although it might have been a gleam in the eye of some of the fibre box producers. We had developed and patented the use of diphenyl and had proven without a question of a doubt that it would prolong the life of citrus. The odor of the diphenyl raised certain questions, including that of toxicity, and we were informed by the principal users that they would not permit its use until this question had been settled, and so

To settle once and for all whether or not the use of diphenyl was attended by any hazard to the health of people consuming diphenyl protected citrus fruits, we chose the Stanford Research Institute to determine this for us. So that the results coming out of this work would be subject to the least criticism by the F.D.A., we instructed the research men to call at the offices in Washington to obtain the advice and recommendations of the F.D.A. on setting up the program so that no points considered important by them would be overlooked.

In view of the complete absence of any cases of poisoning of workers in the manufacturing plants of the chemical companies producing diphenyl, as well as no instance of any trouble from consumers of citrus fruits protected by diphenyl, we felt reasonably certain at the outset that the testing of the toxicological effects of diphenyl in animals would result in data that would give diphenyl a clean bill of health. That this supposition was ill-founded is now a matter of record. The interpretation of the results of the final report issued by the Stanford Research Institute is a highly technical matter that I will not attempt to deal with because it has been discussed by experts this afternoon. However, since the present attitude of the F.D.A. is a reflection of the work done at the Stanford Research Institute, I must refer to the Stanford data to a certain extent. In doing so, perhaps I can give some assistance to Mr. Gibson, the next on the program, on "Where do we go from here?"

The first step in the research program at the Stanford Research Institute was a 90-day feeding experiment with rats in which the test animals were divided into four groups and were fed zero, 0.01, 0.03 and 0.1% diphenyl. During the 90-day period, no appreciable differences developed between the control group and those receiving diphenyl in their diets. Examination of the sacrificed animals at the end of the experiment showed no evidence of tissue degeneration or abnormalities due to toxic conditions. These results were reported to the F.D.A. and they suggested that two-year feeding tests should be made with higher levels of diphenyl in the diet, namely, 0.01, 0.1, and 1%, the procedure that was later adopted.

At that time we did not know the amounts of diphenyl in citrus fruits that were picked up from the diphenyl-treated wrappers. This point was called to the attention of the F.D.A. but they suggested that the feeding experiments be started before this information was obtained. On raising the question that 1% diphenyl was a relatively large amount they stated the desirability of testing a chemical at a high enough level so that toxic effects might be observed. These recommendations were accepted at their face value and only at the completion of the work was it revealed that we had made an error in judgment.

The major part of the program at Stanford consisted of a two-year feeding experiment on four groups of rats given diets containing 0.01, 0.1 and 1% diphenyl, and one-year feeding experiment on monkeys using the same levels of diphenyl feeding. Also skin irritation tests were made on rabbits and reproduction studies were made with rats over several generations.

By the end of 1952 the feeding experiments had been completed although the pathological data were incomplete. On December 8, 1952, Dr. G. W. Newell, in charge of the program at Stanford and Mr. J. W. Linehan, of our office, called on the F.D.A. to apprise them in a preliminary way of the results of the feeding tests. This was done with a full expectation that the F.D.A. would recognize diphenyl as an innocuous substance when used as a chemical in citrus fruit wrap. However, in a letter dated January 16, 1953, Mr. M. R. Stephens, Assistant Commissioner of Food & Drugs, wrote to Dr. Newell as follows:

> "While all the data are not yet available for study, it is the conclusion of our Division of Pharmacology, based on the now available data, that diphenyl is a poisonous or deleterious substance as shown by the chronic toxicity studies."

While this development was unexpected, it gave the Stanford men advance notice of the attitude of the F.D.A. which would be helpful to them in interpreting the final results for the benefit of the F.D.A. We felt that the F.D.A. had not given proper consideration to the quantities of diphenyl that could be ingested by the average consumer of citrus fruit and that when the figures were presented in the final report that there would be a change in attitude. This dangerous situation might have been averted had not a bomb shell been dropped in the form of a publication, The Produce News, of February, 1953, in which the F.D.A. was quoted lto the effect that diphenyl is a "poisonous or delegerious substance unfit for use in or on food and users of the chemical are so notified." We do not know the source of the leak of the information but the effect was to put the F.D.A. on a spot from which there was no graceful retreat. The official denial of the article in The Produce News was published a week later in The Packer in which Mr. Crawford, the Commissioner, was quoted as saying:

> "F.D.A. has made no thorough study of the problem and toxicity studies on the subject were few and nonclusive."

The article then continued:

> "However, it is crystal clear that diphenyl by itself is a poison. On the other hand, it is by no means clear that diphenyl cannot be used witout harm to the consumer when it is used in wraps or containers in the shipping of citrus fruits."

The last two statements in The Packer seem to summarize very concisely the current position adopted by the F.D.A. and reflects the state of uncertainty in which we find ourselves. The F.D.A. has not retreated from its position that diphenyl is "A poisonous or deleterious substance" and they choose to interpret the results of the final Stanford report that diphenyl at an intake level of 0.01% is also toxic. A letter dated August 21, 1953 by Mr. Stephens to Dr. Newell commenting on the final report stated:

> "Our division of Pharmacology has commented that because of similar damage in some of the controls, and in the test animals at the dosage level of 0.01 percent, the effect at that level is questionable. However, as your letter of transmittal to the Crown Zellerbach Corporation indicates, the frequency and intensity of the pathologic changes is greater in the diphenyl-fed group than in the control group. This seems to leave no doubt that diphenyl at the 0.01 percent level either causes the kidney damage or intensifies the changes that normally occur in the older rats."

Then further--

> "If the question of the establishment of a tolerance is formally considered, it is believed that additional chronic studies should be carried out at the 0.01 percent and lower levels in order to demonstrate a level of safety." This is important. "Since experiments in other species have shown little or nothing, we believe you may wish to consider modifying your program to include only a rat experiment at these lower levels. The important point is, if the question of the establishment of the tolerance is formally considered, it is believed that additional chronic studies should be carried out at the 0.01 percent and lower levels. I called on the F.D.A. in Washington with Dr. Newell and found them extremely friendly and co-operative. They wanted to work with the industry, and pointed out very clearly that if they were to take any action to approve a tolerance it would be on the basis of an appeal from growers, packers, or consumers, indicating an economic necessity. An appeal from the Kraft Board Manufacturers, Paper Manufacturers, and Box Manufacturers would be of no use. If a tolerance is to be adopted or set up it must be in the form, of an appeal from the producers, growers, or consumers and on the basis of economic necessity."

Commenting on thhs latter statement of Mr. Stephens, Dr. Van Horn in a letter to our Dr. Moyer put his finger upon a very important point. With Dr. Van Horn's permission I will quote the following from his letter:

> "I most certainly hope I have misunderstood the intent of his proposal, otherwise it is difficult to orient it to the objectivity that should characterize all investigations. If there are species which are impervious to diphenyl it would appear fair to include them in the study and then weigh results from them with those with rats in the light of what might happen in the human being."

We have distributed copies of the final report of the Stanford Research Institute to a number of responsible people among whom are Dr. Van Horn and Mr. Gibson. The appraisal of the scientific data is a matter for experts and the concensus of opinion of those experts that have analyzed the report is that the Stanford Research Institute obtained a "bad" lot of rats for the experiment and were unfortunate in having an outbreak of pneumonia in the colony that in itself was sufficient to throw doubt upon the final results. The opinion further is that there is just as much evidence that diphenyl at the .01% level is harmless. Furthermore, we should remember that at the .01% level, this amount of diphenyl is some 2,000 times the amount that a human being would consume in citrus juice from diphenyl protected fruit.

In summary, the F.D.A. has classified diphenyl as a poisonous or deleterious substance but are not ready to ban its use because of uncertainty as to whether this characterization of diphenyl applies to the amounts found in citrus fruits. They have suggested that if a tolerance is formally considered that additional chronic studies should be carried out at the 0.01% and lower levels in order to demonstrate a level of safety. This question is of joint interest to the citrus growers, citrus packers, and box manufacturers and, as the situation now appears, to only a very limited extent to the tissue paper manufacturers. If it is decided that these further tests should be made, in my opinion they should be made promptly. Possibly Mr. Gibson will answer this question in a discussion on "Where do we go from here?"

WHERE DO WE GO FROM HERE, AND WHY?

Geo. B. Gibson,
Managing Director, Fourdrinier Kraft Board Institute, Inc.

Before commenting on my subject, "Where Do We Go from Here, and Why?", I would like to make a few remarks. I am sure that you are as appreciative as I am of the work the panel has done and the presentations that have been made this afternoon explaining this rather difficult matter to us. I particularly want to tell the members of the staff of The Institute of Paper Chemistry what an excellent job they have done in coordinating all of the work from Florida, California, Monsanto Chemical Co., and Dr. Hazleton and maintaining an objective approach throughout. I think this work is worthy of the highest commendation.

I am not going to try to summarize what has been said this afternoon. I am going to attempt to demonstrate the importance of this subject to us, and my recommendations for its disposition. The subject assigned to me is, "Where Do We Go from Here, and Why?". I'd like to examine the "why" first.

Why is diphenyl important to us? Because we have found, first, that it was impossible to ship citrus fruit economically and safely in corrugated containers without the use of a fungistat; and, second, that the only fungistat which has been successful from the standpoint of controlling decay and stem end rot is diphenyl. Without diphenyl or its equivalent as a fungistat the industry would lose a potential market, which we are at present developing, in excess of 150,000 tons of corrugating board per year. This would equal the tonnage of a mill producing 500 tons per day. Its end product dollar value would exceed $30,000,000 per year.

You have heard the subject of diphenyl thoroughly discussed by expert technicians. Mr. Ticoulat has outlined our current position with the Food and Drug Administration. However, the question before us is not whether diphenyl is toxic. We know diphenyl is toxic. So are salt, sugar, chocolate and many other components of our daily diet when used to excess. The question is at what level we can safely use diphenyl. In the determination of that level, several alternatives are open to us:

First, since the Stanford report has been published and submitted to the Food and Drug Administration, that we do nothing further. Such a course, aside from creating an impression of lack of interest on our part, would undoubtedly make it difficult for us in the future to establish suitably toxicity levels for other fungistatic materials that we might be fortunate to develop. Moreover, such a procedure would very probably result in seizure or in a cease-and-desist order, bringing our activities to a standstill.

Second alternative: that we extend the toxicological research initiated at Stanford Research Institute. It would appear that the suggestion of the Food and Drug Administration is a reasonable one--namely, that of extending the investigation and of reducing it to proper limits so that tolerances can be set and methods established for measuring residues in oranges that can be policed. The toxicological research obviously could be extended either at Stanford Research Institute or through some other qualified agency. It seems to me, however, that such further work should be done at some other agency. There is always an advantage in having an important subject like this checked by at least two qualified organizations. In addition, through the work of the people who have already appeared on this panel, we have developed methods of measuring residues which are reproducible in the field, and which should be acceptable to and usable by the F. & D. A. as a policing means. Therefore, we feel that further toxicological research should be done by another agency and by one which is familiar with the methods which have been developed in these recent months.

Third alternative: that we attempt to find a substitute for diphenyl. We are presently working on this program at the Institute of Paper Chemistry, attempting to find such a chemical, but to date nothing has been developed that looks even remotely practical. California has embarked on a similar program, and we recently contributed $8,000 to the Citrus Industry of California in an endeavor to advance their work on this experimentation.

The financing of the entire diphenyl program is a matter for consideration. Should we seek financial aid from the Citrus Container Institute, the Citrus and Vegetable Container Institute of the Pacific Coast, Crown Zellerbach, the Florida Citrus Industry, the California Citrus Industry--and make a community project out of it? I feel that the responsibility for this falls directly on our shoulders, and that if we attempted to make a community project out of it we would fail because of the divergence of principles and ideas. There is no truer statement than that too many cooks spoil the broth.

In conclusion I would like to make these recommendations:

1. That F.K.I., with the help of Monsanto Chemical Company and in collaboration with The Institute of Paper Chemistry, attempt to evolve a plan that is satisfactory to the Food and Drug Administration, using whatever scientific agencies and counsel seem practical to complete the preliminary work done by Stanford.

2. That both the Florida and California Citrus Industries severally and collectively develop their own programs of economic necessity for presentation to the Food and Drug Administration when, as and if required.

3. That all three agencies--the Citrus Industries of Florida and Citrus Industries of Florida and California, and F.K.I.--work co-operatively toward solving this problem and bringing it to a successful conclusion as expeditiously as possible.

Friday Session

October 23, 1953

Semiannual report . . . . Strange

Engineering studies. Relationship between box compression and material constants . . . . . . Whitsitt

Effect of repeated exposure to humidity on board and box characteristics . . . . . . Root

REVIEW OF
FOURDRINIER KRAFT BOARD INSTITUTE
RESEARCH PROGRAM
April 1, 1953--September 30, 1953

John G. Strange,
Vice President, The Institute of Paper Chemistry

It is our privilege to present another nontechnical review of recent achievements in the research and development program which The Institute of Paper Chemistry is pursuing in behalf of your group. Nine years have transpired since this relationship was initiated and these semiannual meetings have become one of our most pleasant obligations.

Inasmuch as this is an anniversary meeting and in view of the several people who are attending one of these affairs for the first time today, it seems appropriate that we review briefly the objectives which were set forth back in 1944 and some of the highlights of the intervening years.

From the beginning, the broad purpose of this program has been the development of information and procedures which would lead to the manufacture of better and more versatile fiber boxes. It was agreed that any program leading to "better boxes" would have to concern itself with at least four major areas of investigation, namely, the basic raw materials, fabrication practices and technics, the performance of boxes under specific uses, and the development of adequate yardsticks for measuring and evaluating progress in the first three areas. It was hoped, among other things, that progress along these several fronts would ultimately enable the paper mill to predict the boxmaking characteristics of the corrugating medium and liners as they are being made on the paper machine.

One does not have to be intimately familiar with this industry or its technology to realize that these original objectives were indeed ambitious ones. It was quickly obvious that sustained progress depended upon the development of what might be called adequate yardsticks. When this program was initiated there was no dearth of instruments for measuring various "properties" of paperboard and boxes. There was considerable confusion, however, as to the suitability of these instruments and there were different schools of thought concerning their application. Before applying our efforts to the development of still further instruments it seemed appropriate therefore that we should review systematically and critically the existing devices, in order that a better knowledge might be gained of the precision of these instruments, the properties they were measuring and, more importantly, the significance of these properties in terms of industry and consumer practices. This was no small undertaking and early reports which were rendered to your group are evidence of the amount of time and effort that went into this phase of the program.

Having completed our review of the existing instruments, the next task was that of developing new devices for the purpose of filling needs which were not adequately met by existing methods. You will recall that our work on instrumentation could not be carried forward in the isolated laboratory but had to be related continuously to your particular raw materials and to the use requirements imposed by the manufacturers, the shippers, the carriers, and the many consumers of paperboard and fiber boxes. Field studies, therefore, have played an important role in this chapter of your research.

Although we have said so on previous occasions, it is perhaps worth repeating the fact that instrumentation involves more than a study of gadgets. It is the inquiry into the nature and character of materials and products, in order that they may be made more efficiently and used more widely. Properly conceived and applied, instrumentation will reward the manufacturer with improved productivity and the consumer with a more satisfactory product.

A quick review of the instrumentation phase of your program over these recent years indicates certain highlights. First, as a result of considerable study of the functional properties of paperboard or corrugating medium, we now have a much better understanding of the behavior of these materials, their strong points and their limitations. The targets of the manufacturing process have consequently been more clearly defined. Work which has been done on the influence of humidity and moisture, as well as temperature variations, has led to a better understanding of the effects of environment on paperboard and box performance. We believe that it is entirely in order to say that one can predict more reliably and sooner today than was possible nine years ago the ultimate performance of your raw materials. The job is far from finished, however, and as time goes by we continue to learn more and, as we learn, the results can be applied to your practical problems of manufacture and distribution.

Before this program was initiated relatively little co-ordinated work had been undertaken on the relationships between component tests, combined board tests, and tests on the box itself. Thus, in addition to our work on instrumentation, a series of fabrication runs was undertaken for the purpose of systematically varying the characteristics of the components, the combined board, and their integration into the final box. This work has thrown considerable light on the relative importance of such factors as strength and uniformity of the liner, weight and structure of the corrugating medium, and the engineering of the box itself. In addition, these early fabrication runs provided the Institute with sufficient materials to carry on the instrumentation studies and to study the relationships between various properties.

Many of you will recall the baseline study which was made in 1945. At that time the Institute representatives travelled throughout the country impounding rolls of kraft liner in approximately 100 warehouses. Representative specimens were sent to Appleton and these specimens were measured for every conceivable property. Fabrication runs were then made under carefully standardized operating conditions. The resulting boxes were then subjected to extensive experimentation. As a result of this project, the Fourdrinier kraft industry established an objective benchmark of the quality and performance of the board being produced by your membership at that time.

This information proved to be of great value in subsequent discussions with the railroads. It has also provided a reference point against which all subsequent work and progress could be measured.

The original baseline study has been supplemented by other conversion runs, the most recent of these being made only a few weeks ago. In each instance these conversion runs have thrown new light on such specific points as the relationship of weight distribution to box performance, the effect of different types of adhesives, and the relative influence of different types of corrugating medium and flute designs. This accumulating information has modified many opinions, including some of our own, with respect to test and material relationships.

Roughly five years ago a continuous baseline program was initiated. This has provided a running diary of the performance of your industry in so far as linerboard is concerned and has established a basis against which you may continuously evaluate your progress as an industry or as a unit of that industry. One of the significant byproducts of this particular program has been the establishment of what amounts to a calibration system among the various participants, and individual mills may now compare their own test results with a certain degree of confidence against the results of other laboratories.

In addition to the instrumentation studies, the fabrication runs, the continuous baseline program and the various fundamental inquiries which we are making into the nature of your materials and their behavior, there have been several developmental programs. Among these is the development of the citrus fruit box, the work which has been done on fungistats, the work which has been done on masking materials, and such specific undertakings as the development and manufacture of the single fluter. The latter, incidentally, is directly responsive to one of the original objectives of the program, namely, that of being able to predict the potential performance and quality of paperboard as it is being manufactured.

It sometimes appears that our present age has an unusual attachment to numbers. Perhaps this is partly due to the fact that bodies of knowledge are constantly growing and we are becoming more and more specialized in our individual tasks and the things that we make and the services we perform. Very few of us are comfortable in the presence of something we cannot understand or something we cannot evaluate. Our serenity seems to require a means or a system for placing things in perspective in order that we may say "this is good and that is bad" or "I prefer this because ..." And the pace of modern life apparently is such that we do not want to waste too much time in reaching our conclusions. In this emotional and intellectual climate, the statistician has been welcomed with great enthusiasm. He samples, correlates, throws out the "aberrations" and winds up with a series of numbers, or perhaps even a single number, which neatly puts everything in place, and we are no longer tormented by indecision because, after all, we can count, can't we!

A famous astronomer once predicted, in the midst of a popular lecture, that the world would probably come to an end in approximately thirty billion years. As soon as he had finished, an excited member of the audience rose and shouted: "When did you say the world was going to end?" "In about thirty billion years" the astronomer answered. "Thank goodness," the excited person replied, "I thought you said thirty million years!"

Yes, we have an astonishing faith in numbers. The retail trades are keenly aware of this attitude and have demonstrated that $2.99 is frequently more appealing than a straight $3.00, or perhaps even $2.90. The odd figure seems to indicate a greater precision, a more earnest effort to achieve something. There is a flavor of conviction about it.

Numbers provide a kind of intellectual shorthand, as well as a convenient mechanism for lending authority to an arbitrary decision. And sometimes they retain their authority long after the concept which brought them into being has been outmoded. In all aspects of our society we seem to be extremely busy at this business of searchingfor new numbers, new symbols, which will provide a crutch for our thinking or a basis for our decisions. We are engaged in a breathless search for the right formulas--everyone, we might say, from the people who are working on your program at the Institute right through Washington and, finally, to the well-known Dr. Kinsey!

Now there is nothing wrong with numbers themselves and certainly the use of symbols for the expression and integration of complicated concepts has led to achievements which might otherwise have escaped us. We believe, nevertheless, that the unrestrained use of, and faith in, numbers frequently results in the creation of "sacred cows" or spurious axioms whose apparent validity rests on a purely quantitative foundation with little or no justification from the standpoint of fundamental philosophy, quality or substance. There are some good examples of this in the paper industry.

We have, by way of illustration, one rather famous number ordinarily referred to as Rule 41. And this number has, in turn, rested on still another number, or perhaps one should say a series of numbers which are calculated on a device known as the "Mullen tester." We do not mean to disparage the progress which was originally made through the use of the bursting strength tester, nor do we intend to belittle the constructive statesmanship which has been involved over the years in the exercise of Rule 41. We do believe, however, that a rather undue faith was developed in the purely quantitative aspects of the so-called Mullen test, with insufficient attention to the whimseys involved in its execution and the real meaning of its readings in terms of end use. Many of you will recall that our instrumentation studies have demonstrated that there may be differences of as much as 25% in the readings obtained on Mullen testers which have been calibrated under the old procedures. This wide disparity in testing results imposed an unduly severe regime on the industry and, if it were possible to add the unnecessary costs involved in the grim efforts of this industry and the carriers to follow the dictates of the unbridled Mullen test, they undoubtedly would run to millions of dollars. Through your program we believe that considerable progress has been made on this front and experiences of the last several years would seem to suggest that there are fewer disputes between the mill laboratories and among producers and consumers as a result of this work. We know, also, that bursting strength is merely one index of board performance, and sometimes a relatively unimportant one.

In our work here at the Institute we are continuously developing still more numbers--new numbers--in our testing program and in our structural studies. The important thing, however, and the point which we are trying to make, is that one should realize that these numbers have no real meaning or significance unless they are a reliable index of quality and performance. We are seeking, in other words, a qualitative understanding of the industry's manufacturing, converting and material variables and this objective must be constantly in our minds as we review the numbers and quantitative results of various research activities. If this be an admonition, it is aimed fully as much at ourselves as it is our sponsors. Let us not confuse insight with arithmetic.

In the nine years which have transpired since the initiation of your research program, the production of your industry has more than doubled. We are not suggesting that there is any connection between these two circumstances--unless it be that both required a faith in the future and a determination to meet it adequately and vigorously. We do suggest, however, that the growth in your industry provides an even greater validity, and one is tempted to say, urgency, to the objectives which you had in mind when the research program was originally started.

It is elementary to observe that growth is output requires a corresponding growth in input. This industry has developed a tremendous appetite for raw materials and especially for its principal raw material--the tree. As one projects this appetite into the future he becomes increasingly concerned with ways and means of satisfying it, and it would appear as though there are at least two general approaches to the problem.

The first and most obvious approach involves the trees themselves and on this front one sees a great deal of activity. Work which is going forward on insect control, tree farms, reforestation, selective breeding, development of new species, more efficient cutting, use of wood waste, and high yield cooking processes is all very much to the point.

There is a second approach, however, which is not quite so obvious because its results are indirect. This approach involves a more efficient use of the paperboard which you are making from the raw materials. Here we believe your research program at the Institute can play an important part. As more is learned about the structure of the paper box and the properties in your corrugating medium and liner which have a direct influence on box performance, the weight of your materials can be distributed in a more effective manner. As better instrumentation becomes available, it should be possible to produce materials of greater uniformity, thereby eliminating undue safety factors and waste in manufacture. As accumulating information enables us to predict more reliably the ultimate performance and potential boxmaking characteristics of our product we can aim more specifically and more economically at end uses and can promote the more efficient use of board in the converter's plant. Progress on these several fronts can contribute significantly to the growth of the industry <u>and</u> to the conservation of its raw materials.

Referring specifically to the work which has been in progress since last March, the following comments may be of interest.

Long-Range Program

A. During the past six months five new reports have been prepared; three of these have been distributed to your membership and two are now at the printers. The following subjects are involved:

1. A study of the effect of corrugating roll nip pressures on the performance of combined board and boxes.

2. An investigation of the relationship of scoreline to box compression.

3. A preliminary study of the effect of flap score contour on box compression.

4. A study of the relationship between box design and compression characteristics.

5. A study of the effect of ventilating holes on box compression.

B. Work has been completed on two additional investigations and reports are now being written covering this work.

The subjects are:

1. The behavior of combined board and boxes under repeated exposure to humidity cycling.

2. A comparison of the behavior of different corrugating adhesives when exposed to humidity cycling.

C. In progress is work on the following subjects:

1. Engineering studies (for the benefit of those who are here for the first time today, we should say that these engineering studies are aimed at the development of information on the physical constants of your raw material, in order that box performance may be calculated from a knowledge of these constants.)

   a. An investigation of the mechanical properties of combined board and boxes which influence end load compression.

b. A study of methods for the evaluation of scoreline effects on box compression.

2. A study of the factors which may contribute to regression in bursting strength of paperboard.

3. A study of the Concora medium tester.

Developmental Studies

During the past six months we have continued to spend a great deal of time on problems which have arisen through the use of biphenyl in citrus fruit containers. The general scope of this work was covered in detail yesterday and we are sure that you will appreciate the intensiveness of the effort and consequently will view with some charity the costs that have been involved. In addition to the almost endless analyses which have been performed, we have been busy at the development of new masking compounds and also have continued the search for alternate fungicides. Two reports have been distributed to your group on these subjects, one dealing with the masking of biphenyl in oranges and the other dealing with the effect of various chemicals on the growth of organisms attacking citrus fruits. There will be further reports dealing with the analytical work, both as regards methodology and data obtained through the shipping and holding tests.

Another major effort of these recent months has been the fabrication runs which were made at Menasha. There have been 32 conversion sequences and we still have five to go. Obviously we will be busy for quite some time analyzing and interpreting the results. Mr. McKee plans to give a progress report on this subject later today.

Continuous Baseline Study

The number of samples involved in the continuous baseline study continues to grow slowly. The results for the past twelve months have been summarized and will be presented at this meeting. So far as we are concerned, these monthly reports continue to be of considerable interest and we hope that you regard them in the same light. In accordance with your request, a quarterly report is now being prepared summarizing the over-all production of your membership, as well as the production in different grade categories. This information is issued as a supplementary report to the continuous baseline studies. The production data for the third quarter of 1953 will be distributed later today.

Financial Report

Expenditures for the six-month period beginning April 1, 1953, and ending September 30, 1953, may be classified as follows:

$20,684.55 has been expended on long-range research
14,489.65 on the baseline program
34,776.75 on special studies and on the citrus fruit program.

This makes a total of $69,950.95.

We should like to point out that the expenditures for long range research and the continuous baseline program are a little less than the previous six months, but approximately in line with historical experience. The balance of roughly $35,000 has gone substantially into the toxicity work which we and others have been pursuing with respect to biphenyl and into the recent fabrication run. The investment in the biphenyl research, including outlays for outside consultation and services contracted on your behalf by the Institute, amounts to nearly $20,000. The special fabrication run has thus far cost approximately $10,000, and of this $6,200 was paid for materials which were purchased from your own membership.

ENGINEERING STUDIES OF TOP LOAD BOX COMPRESSION

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The utilization of corrugated board in shipping containers rests ultimately upon its competitive price and the ability of the container to safely transport its contents. Consequently, it is of some importance to the board producer to understand what properties of the board influence and control the container's performance in service. Speaking in general terms, this involves both the definition and maintenance of container quality. Because of their importance your organization has sponsored considerable research in the field of container strength and I should like to briefly review recent studies directed toward evaluating one aspect of container quality--top-load box compression.

One simplified manner of visualizing top-load box compression strength is to consider that its resistance to compression is dependent upon two factors. One of these factors is the flap scoreline and the second is the panel wall or tube. The tube, which is merely a four-sided structure without top, bottom or flap scores, makes possible the study of panel wall resistance to compression with no complicating scoreline effects. A number of studies have shown that the tube always sustains a greater compressive load than the box of similar size. Thus, the tube is sometimes said to represent the "ideal" box strength because the presence of the flap scores always diminishes the load sustained by the panel walls. In other words, the flap scores might be likened to a weaker link in a chain.

Past container research has been based in part upon this simplified visualization of box compression--that is, initial work focussed upon the compression strength of tubes (panel wall compression resistance) and, more recently, studies of the compression potential of the flap scoreline have been inaugurated. Before discussing these later studies, I should like to briefly review the studies of tube compression.

In the first studies of tube compression strength, tubes whose dimensions ranged from 3 to 24 inches in depth and from 36 to 68 inches in perimeter were constructed. Test observations indicated that each panel wall of these tubes buckled and failed as thin plates. This suggested that an engineering analysis of thin plate compression strength could be applied to these combined board tubes. Because paperboard properties vary with direction, it was found desirable to treat it as an orthotropic elastic substance. The results of this first analysis of tube strength were such that estimated loads were within 10% of the observed loads in about 80% of the comparisons. This original analysis was simplified in a later report with avout the same precision of estimate. In general, the simplified analysis indicated that the compression strength of tubes depended upon their dimensions, the stiffness and tensile or compression strength of the board. On an over-all basis, the results indicated that worthwhile estimates of tube strength were obtained from this approach.

Attention now centered upon the effect of the flap scorelines on top-load box compression. As a first approach to the problem, boxes and tubes were constructed in our laboratories. Depth dimensions varied from 5 to 24 inches and in perimeter they varied from 44 to 68 inches. This chart illustrates the trends of box and tube compression with depth for one of the perimeters. One of our earlier remarks is illustrated here because the box strength is in all cases considerably less than tube strength due to the effect of the flap scorelines. It may be noted that as the depth decreases the box strength increases much more slowly than tube strength. Thus, the ratio of box to tube strength varies with depth. At small depths the boxes seem to sustain a considerably smaller per cent of tube load than at the greater depths. In fact, for all the data, box strength varied from 44 to 71% of the corresponding tube strength. It was concluded that the effect of the flap scores on box compression varied with box dimensions.

As part of these efforts we have been investigating the merits of a direct test of the compression potential of the scoreline. What we have done is to test a short column--one end of which contains the flap score area. As might be expected, tests have shown that the strength of the column is considerably reduced by the presence of the scorelines. In our initial efforts to utilize these scored column results, they were correlated together with box results in the same type equaations developed in our earlier tube work. The box data, illustrated in part in the preceding chart, were used and the equations seemed to fit the data well. As noted in Report 39, about 90% of the estimated box loads were within 10% of the observed box loads.

Because these results seemed promising, further efforts were made to test their utility during these last six months. In one study, fourteen sets of B-flute, No. 2-1/2 can size boxes were evaluated for top-load compression and for scored column strength. These results were then statistically correlated together to give a quick but crude estimate of the strength of the relationship between the two tests for different materials. As may be seen in this chart, the scatter of the points indicates that the relationship between the two tests is not a perfect one which might be expected in view of the many variables that probably affect box compression. The correlation coefficient--which is a relative measure of the strength of the relationship--was 0.83 which is of intermediate magnitude but indicates some definite correlation between the tests.

While the above testing was in progress, a small investigation of the effect of scoreline type and severity on box compression was undertaken. For these studies, boxes were fabricated from two samples of A-flute board utilizing two types of flap score wheels. One board sample was fabricated with 42-lb. liners and the other was fabricated from 69-lb. liners. One set of boxes from the stronger sample was also fabricated using a lighter score--that is, a greater distance between scoring wheels was utilized. For each scoring condition boxes were constructed and tested having three perimeters and three depths. Composite averages of the box data are shown in the chart. For example, it may be noted that the score 2 boxes gave smaller loads for Sample A and only slightly greater loads for the stronger

Sample B. With regards to the effect of score severity it may be noted that those boxes fabricated with the lighter score seemed to give smaller loads than those fabricated with the heavier score.

Scored column tests were also performed on these materials and this chart illustrates the precision of estimating box loads from the second column data and plate equations for the above data. It may be noted that the estimates of box strength are reasonably good as about 75% of the estimate are within 10% of the observed loads. In a few instances discrepancies as great as 16 or 17% may be observed. These estimates are based upon a correlation of all box-scored column data obtained thus far and would seem to have promise. Some caution should be exercised, however, because we have found instances where the scored column and box tests contradict each other--that is, when the boxes lose strength the scored column test is actually greater. Another difficulty arises because the variability of the scored column test is often larger though this may be one cause of the reversals mentioned above. In any event we are making efforts to locate the chief causes of such variability and reduce them where possible.

In conclusion, I have tried to summarize briefly the results of our current work on top-load box compression. You will note our first efforts centered on obtaining an understanding of those factors that affect tube compression strength. This was followed by our current efforts to investigate the effects of the flap scoreline on box compression. In that work we have tried to develop a direct test of the scoreline itself--the scored column test--and to relate this test to the actual performance of boxes through such variables as type of score, severity and material. The results seem promising thus far, however, further effort seems necessary to improve the scoreline test and to understand the relative effect of the various scoreline variables.

THE EFFECT OF REPEATED EXPOSURE TO HUMIDITY CHANGES ON BOARD AND BOXES

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It is quite well known that boxes stored under high humidity conditions will pick up moisture and lose some of their stacking strength. For example, using conditions of 50% relative humidity as a baseline, top compression has been observed to increase 9% when tested at 35% and decreases 10% when tested at 65%. End compression has been observed to increase 2% at 35% relative humidity and decrease 19% when tested at 65% relative humidity. While this change is generally known, it has been the feeling that if a box were subjected to increased or decreased humidity and then properly reconditioned to 50% conditions, the strength would return to the original value.

Recent experiments with wetting and drying corrugated board repeatedly and testing for changes in bonding of the liners have indicated that the adhesion on some materials has greatly deteriorated. When it is realized that a box has load bearing characteristics as a result of its engineering structure wherein the liners are held apart by the corrugating medium and the bond between the liners and the medium, it seems reasonable that if the bond may be changed by changes in moisture content, the strength of a box may also be changed.

It is inevitable that during the period between fabrication and ultimate use as a box, the corrugated board will be exposed to daily or even hourly changes in the relative humidity of the atmosphere so that moisture is picked up and given off from the board. The question then arises, does this fluctuating moisture content adversely influence the board strength? In an effort to answer this, boxes and board were obtained as soon as possible after manufacture and subjected to regular cycles of exposure to high and low relative humidity and sampled at each cycle and conditioned at 50% relative humidity for testing. In addition some material was dipped in water and then carefully dried out and reconditioned prior to testing to observe the effect of water on the strength.

The box compression tests indicated that strength was lost as a result of repeated cycling of humidity as shown:

	Box Compression		
Cycles	Top Load	End Load	Moisture
0	965	560	7.93
1	865	605	7.97
2	840	550	7.87
3	830	690	8.06
4	820	625	7.93

Another set of boxes which had been dipped in water momentarily and then dried and conditioned gave:

	Top Load		End Load	
Control	515		650	
1 cycle	445	-13.6%*	580	-11% *
Water dipped	415	-19.4%*	385	-41% *

Combined board which had been momentarily dipped, dried, conditioned, and tested gage:

	Basis Weight	Caliper	Burst		Flat Crush	Tear in	Tear across
Control	138	0.206	198	1.435 *	28.2	269	235
Water dip	141	0.206	229	1.62*	25.4	326	279

* In red.

Tests were made on some 32-lb. kraft board volunteered by one of the co-operators, to see whether the board properties themselves were changed or whether the corrugated board changes occurred solely as a result of changes in adhesion. The samples of board were cycles between 35 and 85% R.H.

Caliper--increased	2%/cycle	Ring Crush	increased very slightly	
Density--decreased	1/3%	Adhesion	increased	1%
Moisture--increased	.15%	Basis Wt.	slight increase	1/3%
Mullen--increased	2%	E	decreases	3%
Stretch--increased	3%			

A further test was made in tension by loading a sample to failure and obtaining the stretch and the ultimate load. A second test was made in which the sample was preloaded to 7/8 the ultimate load, unloaded, and then reloaded to failure to obtain a mechanical conditioning. A third test was made as a repeat of the second except that the sample was placed at 85% R.H. for 3 days and then preconditioned, conditioned and tested to failure.

	Stretch	Load
1st test	5.28%	38.75
2nd test	2.81%	38.13
3rd test	4.16%	37.54

These tests have tended to indicate that the history of the board and boxes between fabrication and final use may have a significant influence on the physical properties of the finished container. It is apparent that if the full potential is to be realized from the box some attention should be given to the conditions of storage between fabrication and use.